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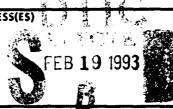
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6. AUTHOR(S)

Eric Mazur

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Harvard University
Division of Applied Sciences
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Cambridge, MA 02138



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13. ABSTRACT (Maximum 200 words)

The thrust of this project was to study the vibrational dynamics of infrared multiphoton excited (IRMPE) molecules in a free supersonic jet expansion using time-resolved broadband coherent anti-Stokes Raman spectroscopy (CARS). The work carried out is an extension of work previously carried out under ARO contract DAAG29-85-K-0060 which expired in February 1988.

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Coherent anti-Stokes Raman spectroscopy of Highly Vibrationally Excited Molecules

Final Report

Eric Mazur

October 16, 1992

U.S. Army Research Office

DAAL03-88-K-0114

Harvard University

Approved for public release; Distribution unlimited.

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FINAL REPORT

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5. NAME OF INSTITUTION:

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6. AUTHOR OF REPORT:

Eric Mazur

7. LIST OF MANUSCRIPTS SUBMITTED OR PUBLISHED UNDER ARO SPONSORSHIP DURING THIS REPORTING PERIOD, INCLUDING JOURNAL REFERENCES:

- a. "Energy localization in infrared multiphoton excited CF₂Cl₂ studied by time resolved Raman spectroscopy," Jyhpyng Wang, Kuei-Hsien Chen and Eric Mazur, Int. Conf. Quantum Electronics 1988, Tokyo Japan, Techn. Digest, 496
- b. "Can chemical reactions be controlled with picosecond infrared pulses?", Eric Mazur, J. Electr. Chem. Soc. 35:C386 (1988)
- c. "Multiplex coherent anti-Stokes Raman spectroscopy study of infrared-multiphoton-excited OCS," Kuei-Hsien Chen, Cheng-Zai Lü, Luis A. Avilés, Eric Mazur, Nicolaas Bloembergen and M.J. Shultz, J. Chem. Phys., 91 (3) (1989) 1462
- d. "Multiplex CARS Study of Infrared-Multiphoton-Excited OCS," Kuei-Hsien Chen, Cheng-Zai Lü, Eric Mazur, Nicolaas Bloembergen and M.J. Shultz, to appear in Laser Spectroscopy, Ed. M. Feld (Plenum, 1989), 439
- e. Collisional and intramolecular dynamics of low-lying vibrational states of infrared multiphoton excited molecules, Eric Mazur, Cheng-Zai Lü, Shrenik Deliwala and Jay Goldman, Tech. Digest. XVII Int. Conf. on Quantum Electronics 8 (1990) 214.
- f. Coherent anti-Stokes Raman spectroscopy of highly vibrationally excited molecules in a jet, Eric Mazur, Cheng-Zai Lü, Shrenik Deliwala, Jay Goldman, Proc. XII Int. Conf. Raman. Spectr. (Wiley, New York) 232.
- g. Nonlinear spectroscopy of infrared multiphoton excited molecules, Eric Mazur and Cheng-Zai Lü, in Resonances, a volume in honor of the 70th birthday of Nicolaas Bloembergen, Eds. M. Levenson, E. Mazur, P. Pershan and Y.R. Shen (World Scientific, Singapore, 1990) 165.
- h. Direct evidence for n1-mode excitation in the infrared multiphoton excitation of SO₂, Cheng-Zai Lü, Jay Goldman, Shrenik Deliwala, Kuei-Hsien Chen and Eric Mazur, Chem.

Phys. Lett, 176 (1991) 335.

- i. Kuei-Hsien Chen, Cheng-Zai Lü, Nicolaas Bloembergen, and Eric Mazur, J. Raman Spectroscopy, 21 (1990) 819.
- j. Time-resolved spectroscopy of vibrational energy transfer in infrared multiphoton excited SF₆, S. Deliwala, J. Goldman, K.H. Chen, C.Z. Lü and E. Mazur, in preparation.
- k. Coherent anti-Stokes Raman spectroscopy of infrared multiphoton excited molecules, K.H. Chen, C.Z. Lü, J. Goldman, S. Deliwala, and E. Mazur, in preparation.
- 8. SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT AND DEGREES AWARDED DURING THIS REPORTING PERIOD:

Prof. Eric Mazur — half period
Mr. K.H. Chen — half period
Mr. S. Deliwala — half period
Mr. J. Goldman — summer
Mr. J. Wang — quarter period

Mr. J. Wang was awarded the Ph.D. degree in September 1988 on a thesis entitled "Intramolecular energy distribution of infrared multiphoton excited molecules". After a two-year postdoc at MIT he has accepted a faculty position in Taiwan.

Mr. K.H. Chen obtained the Ph.D. degree in September 1989 on a thesis entitled "Coherent anti-Stokes Raman spectroscopy of highly vibrationally excited molecules". He is currently holding a position at General Electric.

9. REPORT OF INVENTIONS (BY TITLE ONLY): None

Eric Mazur Department of Physics Harvard University Cambridge, MA 02138 DTIC QUALITY INSPECTED 3

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FINAL REPORT

 The thrust of this project was to study the vibrational dynamics of infrared multiphoton excited (IRMPE) molecules in a free supersonic jet expansion using time-resolved broadband coherent anti-Stokes Raman spectroscopy (CARS). The work carried out is an extension of work previously carried out under ARO contract DAAG29-85-K-0060 which expired in February 1988.

With the funds allocated under this contract we have a) obtained detailed information on the infrared multiphoton excitation in OCS, SO₂, ethylene (C₂H₄) and SF₆, b) built a supersonic molecular beam apparatus, and c) developed laser and UHV equipment for a new phase in our research program. See points 2-4 below for more information.

2. As originally planned we studied the dynamics of highly vibrationally excited molecules. Below is a brief summary of research findings for each of the molecular systems studied.

OCS. The OCS molecule has three vibrational degrees of freedom. The overtone of the v_2 mode at 527 cm⁻¹ can be pumped by the CO₂ laser. Because of the cross-anharmonicity of the two modes, the population in the v_2 mode can be monitored by observing the coherent anti-Stokes signal from the v_1 mode at 859 cm⁻¹. In bulk OCS it was found that after excitation with 100-ns CO2 laser pulses the distribution within the v_2 ladder is nearly equilibrium at all but the highest excitation. In fact, even though the laser only populates the first overtone level (v = 2), the distribution equilibrates extremely rapidly because of collisions. This rapid equilibration only occurs mostly within the v_2 ladder — very little transfer of energy to other modes (e.g. the v_1 mode) is observed. Thus, OCS provides a convenient way to study the interaction of a nearly isolated anharmonic oscillator with monochromatic infrared photons. In the bulk the excitation is mostly collisional. From the time-dependence of the peak intensities in the CARS spectrum we were able to determine the collisional relaxation of the energy in the v_2 mode.

 SO_2 . The study of infrared multiphoton excitation of small polyatomic molecules such as SO_2 has received considerable attention in the literature. This molecule is representative of small size

systems which, according to the quasicontinuum model, can be difficult to excite to high vibrational levels since the density of states at low excitation is relatively small. While observation of inverse electronic excitation in SO₂ clearly demonstrated that infrared multiphoton excitation to high vibrational levels is possible, these studies left unresolved the issue of which mode of the molecule is actually excited by the CO₂-laser. The ambiguity arises primarily because there is no available CO₂-laser line resonant to the n1-mode. The 9R(32) line is a strong CO₂-laser line close to n1-mode resonance; the 9P(32) line, on the other hand, is resonant with the first overtone of the n2-mode; and the 9R(22) line, one of the more intense CO₂ lines in the 9- μ m region, lies between these two bands (71 cm⁻¹ detuned from v_1 and 44 cm⁻¹ from 2 v_2). Because infrared multiphoton excitation is intensity dependent for a small molecule like SO₂, the strong 9R(22) line is historically the one most often utilized in the literature. It is not immediately clear, however, whether the n1 stretching mode or the 2 v_2 overtone band undergoes infrared multiphoton excitation when this line is used.

Using the time-resolved CARS setup we are able to directly observe the vibrational population in the various modes of the molecule. We determined that the v_1 -mode is excited, and that no detectable overtone pumping occurred under a large number of conditions, including tuning to the 9R(32), the 9P(32), and the 9R(22) CO2-laser lines, samples in a static gas cell or a supersonic jet, and energy fluxes up to 3 J/cm^2 .

It should be emphasized that our results show that both 9R(22) and 9R(32) pumping of SO_2 leads to excitation of the v_1 -mode at 1151.3 cm⁻¹. While these lines are significantly detuned from the band head, they fall near the peak of the rotational Q-branch of the v_1 -mode at room temperature. This result should provide a clearer understanding of previous and future work in the field, and also illustrates the utility of the multiplex BOXCARS technique for direct observation of mode-selective excitation.

 C_2H_4 . Rotational CARS investigations of IRMPE excited ethylene revealed a richly complex structure which was complicated by the fact that CO2-laser pumping directly populated the probe mode. Although we were able to observe several line dependencies on IR excitation, we discontinued our efforts on this molecule — it appears to be too complex to model or understand in sufficient detail at this time. Possible future investigations of this system should probably incorporate simultaneous rotational and vibrational spectroscopy, so that the complete state of the system can be identified.

 SF_6 . Detailed investigation of IRMPE in SF₆ has settled some important issues. We first probed the SF₆ molecules in a pulsed supersonic expansion jet at variable distances from the nozzle to achieve between 10 and 10^{-2} collisions per 100 ns (the time scale of the pump-probe delay). Spectra with variable pump-probe delays were time resolved on a Hamamatsu C1953 streak camera. Near the nozzle we observed extremely rapid redistribution of energy of low lying states due to frequent collisions with molecules excited to the quasicontinuum. Farther from the nozzle, reduced collisions allow longer observation of nonequilibrium excited low-level states. This allows us to gain new quantitative detail about multiphoton excitation near the quasicontinuum threshold and sheds new light on the interpretation of results from previous researchers in the field. We then performed a second experiment in which the collision rate was varied by adding a buffer gas of either Argon or Helium to reduce collisions between SF₆ molecules. As expected we observed a qualitatively similar dependence of the molecular dynamics on the collision rate in the two experiments.

Using a specially developed computer simulation program developed during this contract period, we have been able to conclusively show that a multi-temperature distribution is produced by the infrared excitation of the SF₆ molecules. We are able to approximate this distribution with a two-temperature model, and thus observe the evolution of two temperatures as we change experimental parameters such as collision rate or pump-probe delay. It has become clear that it is collisions, not intramolecular vibrational relaxation, that dominate the dynamics of energy transfer from the laser-pumped mode to non-pumped modes. Analysis of the data yield approximate vibrational state populations of the excited molecular ensemble as a function of time. Interestingly, we find a non-thermal distribution of energy in a laser pumped mode which persists for up to 400 ns after excitation — a time much longer than any intramolecular relaxation time scale. Measurement of the relative excitation in the pump and heat bath modes as a function of delay time and collision rate suggests that intramolecular vibrational relaxation may actually be much slower than previously expected. This important finding may in part account for the wide disparity of previous results in this type of experiments.

3. Supersonic molecular beam apparatus. With funds from this contract we developed and built a supersonic molecular beam apparatus. The adiabatic cooling of the molecules in the beam expansion has two effects: a) the collisional rate of the molecules is greatly reduced because of the translational cooling, and b) far fewer rotational and vibrational states are populated, greatly

simplifying the analysis of the data. Pure rotational CARS spectra of N_2 in the beam show that at a distance of 20 mm from the nozzle the rotational temperature is reduced to below 5 K. At this temperature only the J=0 and J=1 states are significantly populated. Similar low temperatures have been observed for other molecules.

4. Preparation for future work. During a final no-cost extension period of this contract, which served as a bridge until a new contract was obtained, we terminated the current research effort on nanosecond nonlinear spectroscopy of highly excited molecules and made preparations for our experimental goals in the next three-year contract period. We intend to utilize the expertise of our group in nonlinear spectroscopy and surface, molecular and interface physics to study a variety of ultrafast dynamical systems. With molecules, we hope to extend the pioneering work of Zewail and Sorokin in molecular photoexcitation. In particular we plan to investigate molecular clusters and monolayers adsorbed on surfaces. This requires on one hand a new tunable femtosecond laser system and an ultrahigh vacuum chamber. Construction of both of these pieces of equipment is nearly complete.

Ti:sapphire laser development. The laser requirements for the planned research are a highly stable, sub-100-fs pulse oscillator which is broadly wavelength tunable. To this end we have designed and built a self mode-locked Ti:Sapphire laser with high quality, stable optical mounts and components, using a HITCI dye-jet saturable absorber and SF14 prism pair which provides a very stable 100-MHz mode-locked train of 100-fs pulses. We can obtain powers up to 1W of near-TEM₀₀ output in a range tunable from 700 to 900 nm with a birefringent tuning plate and a single set of optics, and a peak slope efficiency of 7% around 800 nm. We have also observed self mode-locking without the saturable absorber jet, which produced ps pulses. We expect soon to complete construction of a 10-Hz Nd:YAG laser pumped multipass Ti:Sapphire amplifier. Pending the availability of funding for a new pump-laser, we are also considering at least two possible new designs at this time for a high-repetition rate (>1 kHz) Ti:Sapphire amplifier.

UHV chamber development. Our sample chamber requirements are unusual for a UHV apparatus. We need a UHV (10⁻¹¹ torr) surface science chamber that allows sample preparation and characterization; however we also require wide angle optical access to the sample with small distances between the sample and the chamber windows, precise multi-axis translational and angular sample positioning, low temperature (10-70 K) cooling, and extremely high

resistance to sample vibration. After visiting several other research labs which have designed chambers for similar purposes, Dr. W. Mieher has designed a two level system which should satisfy all our needs. An upper surface science level will contain ion guns and gas dosers for sample preparation, and LEED/Auger and mass spectrometry for sample characterization. Additional ports on this level will allow optical experiments to be performed simultaneously with surface science probes if desired, plus possible future addition of a load lock for sample changes without breaking vacuum. The lower level will be a small six inch cube with windows on four sides, which allows excellent wide angle, multiplanar optical access to the sample. The sample will be positioned by a large bore vertical manipulator which can be filled with LN₂ or LHe for direct sample cooling, and which can be solidly gripped from side ports to provide precise repeatable sample positioning and high vibrational stability. Pumping will be provided by a turbo pump on the upper level and an ion pump with a LN₂-cooled Ti sublimator below the system. Construction of this system is well underway and we expect to begin experimenting with samples in UHV by the end of the year.

5. Other achievements. Another noteworthy achievement is that the principal investigator of this contract, Prof. Eric Mazur, obtained tenure at Harvard during this contract period. He was also elected Fellow of the American Physical Society. Two of the graduate students involved in the project obtained their Ph.D. degree.

EQUIPMENT PURCHASES

All transactions on this grant were charged to Harvard account 44-935-7237-2-30. A complete list of equipment purchases for this account, listing vendors, items purchased and purchase price, is attached.

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Transaction Summary Report 44-935-7237-30

Page 2

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5/23/89	12	i	B253-70 Y2-1025-	ASER CORPORATION 70R130T BEAMSPLITTER. i* > P-POL. 50 MW/CM-SQD. 10 MS 2ND SURFACE AR COATING HIGH POWER LASER MIRRORS. 10 MS. 10 HZ. FLAT DICHROIC BEAMSPLITTER. 1* 45 DEGREE. P-POL. 50MW/CM-	(0.25°, 45 5, 10 HZ, F 50 MW/CM-9 X 0.25°,	S DEGREE CLAT + SQU.
			íotal	FLAT + 2ND SURFACE AR COAT		
6/01/89	13	8 3 1 3 2 1 1	TSX-IA ID-0.5 MH-2P RSX-I ISX-IB 360-90 910 AC-2 LH2-T LH-I 600A-3 RSA-2 KPC043.A KPX187AR KPX118AR KPX118AR KPX118AR KPX079AR KPX094AR	RT CORPORATION BASIC TRANSLATION STAGE IRIS DIAPHRAGMS OPTIC INSERTS COMPACT ROTATION STAGE BASIC TRANSLATION STAGE 90 DEGREE MOUNTING BRACKET SPATIAL FILTER ADJUSTABLE-RADIUS CHUCK. TO V GROOVES FOR THICK OPTICS LENS HOLDER LENS MOUNT KINEMATIC OPTICAL MOUNT FOR THE STAGE PLANO-CONVEX LENS. FL 100 PLANO-CONVEX LENS. FL 100 PLANO-CONVEX LENS. FL 38 IN PLANO-CONVEX LENS. FL 38 IN PLANO-CONVEX LENS. FL 36 IN PLANO-CONVEX LENS. FL 3	TIPS WITH E OR 3° DIA 0 MM, + AR MM MM MM MM MM MM	BROAU DPTICS
6/21/89 9/07/89	13 13	24382 25549 1		RT CORPORATION VILLE LUMBER & SUPPLY LUMBER & SUPPLIES NOT TO I	96.00 38.99 EXCEED \$100	7

Total SUPPLIES PRINT & STA 4.771.79

.00

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fransaction Summary Report 44-935-7237-30

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Period: 07/01/1988-09/30/1992

keport Date: 16 Oct 92

derer: PETER SAETA

mm. or xn Date	Clas	s PO #	Vendor		Spent	Committed
/19/89	12	24483 25	INMAC MODEL 72 DATA CA	ARTRIDGES	469.41	
			fotai EQUIPMI	ENT	469.41	.00
/01/88	47	23094 4	HARDWARE HOUS	SE INC E. 100NS. LOW PRO	1.304.80 FILE SIMM	
/02/89	47	24484 5	MAC PRODUCTS MAC+ TO DIN	USA O IMAGEWRITER II.	91.90 8 PIN DIN TO	8 FIN
			fotal COMPUT	ER EQUIPMENT	1.396.70	.00
tal for	Orde	rer: PE	TER SAETA		1.866.11	.00

fransaction Summary Report 44-935-7237-36

Period: 07/01/1988-09/30/1992 keport Date: 16 Oct 92

derer: SHRENIK DELIWALA

mm. or						
·xn Bate	Class	PO #	Vendor		Suent	Committed
	i2 -	32213 3 1 2 2 1 1 1 1 4	CVI-EA PTS3-073 PTS3-103 PTS3-103 PR 80-95 PR80-95- AR1-1037 PLCX-25.	AST FLAT MIRROR FLAT MIRROR FLAT MIRROR	3.747.12 R/ARSM 103	7- . 1M
3/26/91				PRODUCTS SF10 EQUILATERAL PRISMS. S		
3/26/91	12	2	C43.493	SCIENTIFIC COMPANY SF18 EQUILATERAL PRISMS. 19 HIGH QUALITY RETRO REFLECT ACCURACY 5 SECONDS	5MM X 15MN	URE = 1°
B/27/91	i2	34846 1	CVI-E	AST BFT. 1T = 0.5MM. DIA 1° OP THE PLANE OF THE PLATE	222.48 TIC AXIS LI	
9/11/91		2	C43.493	D SCIENTIFIC COMPANY SF18 EQUILATERAL PRISMS. I HIGH QUALITY RETRO REFLECT ACCURACY 5 SECONDS	SMM X 15MM	URE = 1°
1/06/91		/ 2 3 1 1	338066 338223 338 2058 133 102 338 261 080114 080136	ER SCIENTIFIC MR 50 LB TRANSLATION STAGE EQ 02 EXTERNAL BRACKET (EQ BASEPLATE Pbq5s P046 PRISM TABLE W/CLAMP BRIDLES. BR3 M.3 BY 6 M.4 BY 6 M.5 BY 8		
1/19/91	12		MODEL 72 HODEL LP	ROPHYSICS CORP IR ELECTROVIEWEK INFRARED LONG PASS FILTER FILTER HOLDER	1.286.50	
1/19/91	12			D SCIENTIFIC COMPANY CONCAVE MIRROR OF FOCAL LE B 12MM		R 24MM.

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Transaction Summary Report 44-935-7237-30

Period: 07/01/1988-09/30/1992 Report Date: 16 Oct 92

|rderer: SHRENIK DELIWALA

rotal for Orderer: SHRENIK DELIWALA

omm. or rxn Date	Clas	s PO #	Vendo	P	Spent	Committed
2/02/91	12	36185 4 4 4 4	NLINGS 133010 133151 338204B 338262	ER SCIENTIFIC SL 25.4 MIRROR/BEAM SPLITT! MOUNTING PLATE BASE PLATE #PB8 TO GO WITH BR4 BRIDLES MA 12MM LONG SCREWS	2.819.54 ER MOUNTS.	
.2/17/91	12	1	RL1024DA	ELECTRO-OPTICS STANDARD UNTAPPED 1024 PIX EVALUATION BOARD	675.00 EL D SERIES	,
)1/07/92	12			NAL INSTRUMENTS NB-DMA2800 COMPUTER, NI 48 & SOFTWARE		
)1/17/92	12	7	LF05B	RT CORPORATION SEE CAT# INFRARED SENSOR CAKU	548. 75	
		2 2 2	W1551000B W155500B W12D500B	(NDUSTRIES INC 5V DC. 1 AMP 15V DC. 500MA +/- 12V DC. 500 MA		
02/11/92	í2	36682	WALL : Yotal	INDUSTRIES INC EQUIPMENT	222.54 13.838.21	•00
02/05/90	13		DPD201	ES DISTRIBUTION SALES AMPLIFIER AMPLIFIER	242.90	
			ìotal	SUPPLIES PRINT & STA	242.90	•00
12/06/89	12	26606 1	LASER	SCIENCE REPAIR OF HANS GREEN EISEN	200.00 CO2 LASER	
			ľotaj	EQUIPMENT REPAIRS/MAINT	200.00	•00

14.281.11

.00

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ATTACHMENT I: Equipment Purchases

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38,305,47

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fransaction Summary Report

44-935-7237-30

Period: 07/01/1988-09/30/1992

Report Date: 16 Oct 92

Orderer: WALTER MIEHER

Yotal for Orderer: WALTER MIEHER

Comm. or Irxn Nate				r	Spent	Committed		
				OMICRON ASSOCIATES 15.661.20 LEED/AUGER SYSTEM INCLUDING: SPECTALEED ELECTRON OFTICS SYSTEM LEED POWER SUPPLY. MODEL NGL10 AUGER ELECTRON GUN POWER SUPPLY. MODEL NGE35 AUGER RAMP GENERATOR. MODEL RGL20 AUGER UNIT. MODEL MUL CABLES. MANUALS				
		1		REFERENCE OMICRON QUE 9 \$35.605.00 LESS NSF PYI 12% DISE ELECTRICAL CONNECTION	דאטס:			
03/13/92	12	36902 1	UTI	12.500.00 QUADRUPOLE MASS SPECTROMETER SYSTEM INCLUDING UTI 100C-30 MASS ANALYZER. MODEL 03910-1111 CABLE. PART #05168-0010 RF/PROBE CABLE. PART #05185 @ \$14.500.00 LESS \$2.000.00 UNIV DISCOUNT				
∂8/07/92	i2 36903 1		OMICR	OMICRON ASSOCIATES 10.144.27 LEED/AUGER SYSTEM INCLUDING: SPECTALEED ELECTRON OPTICS SYSTEM LEED POWER SUPPLY. MODEL NGL10 AUGER ELECTRON GUN POWER SUPPLY. MODEL NGE35 AUGER RAMP GENERATOR. MODEL RGL20 AUGER UNIT. HODEL MUL CABLES. MANUALS				
		i		REFERENCE OMICHON QUO @ \$35.605.00 LESS NSF FYI 12% DISC ELECTRICAL CONNECTION	OUNT			
			fotal	EQUIPMENT	38,305,47	7		